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DETERMINATION OF DISPERSION IN ANISOTROPIC MICA AND WOLLASTONITE PARTICLES USING THE LAYER SEDIMENTATION METHOD

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The results of studying the dispersion composition of muscovite mica and wollastonite, whose particle shape is significantly anisometric, are described. A simple method for sedimentation analysis is proposed, which makes it possible quickly and with great accuracy to determine the particle distribution based on their sedimentation diameters. Relationships are obtained which can be used to estimate the geometrical sizes of particles for the considered materials.

The use of new composite and mica-ceramic materials in various sectors in economics expands the application area for finely dispersed mica. A promising direction is the application of muscovite mica as the potassium-bearing flux in the production of certain kinds of porcelain. The consumption of wollastonite in the world industry is expanding. Whereas it 1996 is amounted to about 600 thousand tons, by the year 2005 this quantity is expected to grow 2.5 times. The share of wollastonite consumption in production of ceramic, heat-insulating, and construction materials is around 60%. Therefore, it is very important to know the granulometric composition of mica and wollastonite concentrates.

It is rather difficult to determine the granulometric composition of disperse materials, including mica and wollastonite, in which the particle shape significantly differs from the regular volumetric figures. Sieve screening of such materials is virtually impossible, and the microscopic analysis is extremely labor-consuming, especially in measuring needle-shaped particles, since such particles have to be classified not by one size (a described or inscribed circumference diameter) but two sizes (the length and diameter of needles). The use of numerous up-to-date methods for dispersion analysis employing laser diagnostics leads to significant errors (by over 50%), since the analysis determines the volume of a particle assumed to be spherical, and the segment of a needle visible beneath the scanner ray from different angles is taken as the characteristic size. The conductometric analysis oriented to volume measurement, in spite of large samplings, cannot ensure a sufficient precision either. The sedimentation method is limited by the rigid requirements imposed on the shape of the particles, as the formula estimating the liquid medium resistance to a moving particle used in this method (the Stokes formula) was derived for spheres. However, the sedimentation methods of analysis are especially interesting for practical specialists involved in heat- and mass-transfer processes, because unlike other methods, they take into account the hydrodynamic characteristics of motion.

The methods most commonly used in practice are the volume sedimentation methods [1], although due to the uncertainty of the initial moment and the difficulties in processing the accumulation curve, inaccuracies are possible. The analysis of materials in this study was carried out employing a VS-3 weighing sedimentometer developed at the Research Institute of Applied Mathematics and Mechanics, which implements the principle of starting-layer sedimentation, making it possible to analyze materials in a wide range of densities, sizes, and particle shapes with a high precision (USSR Inventor's Certif. No. 1226175, RF patent No. 2000563).

A detailed description of the measuring device and its operating principles is given in [2]. However, it should be specifically noted that the size of settling particles is calculated not only based on the Stokes law [2] but also based on other, more precise laws in accordance with the sedimentation rate. The percent content of each size fraction is calculated on a computer in relation to the total weight of the

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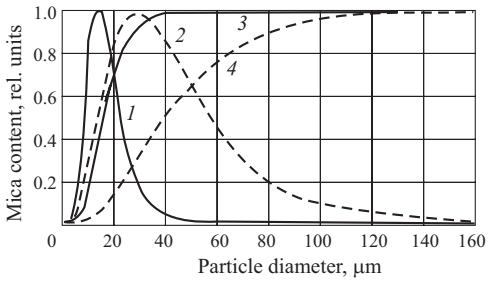

Fig. 1. Distribution of dispersion composition of mica: *1* and *2* and *3* and *4* are, respectively, differential and integral curves of sedimentation (*1*, *3*) and microscopic (*2*, *4*) analysis.

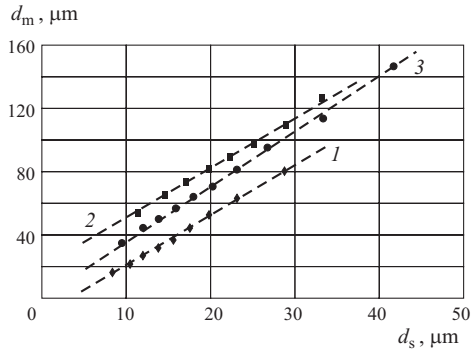

Fig. 2. Correlation between sedimentation d_s and microscopic d_m diameters for various dispersion compositions of muscovite mica: *1 – 3* are trend lines; the symbols designate experimental data.

given sample; therefore, there is no need for precise weighing of the sample before the analysis.

Numerous parallel experiments revealed that the mean quadratic deviation in determining the particle size does not exceed 3 – 5%.

The present study considered the following aspects:

– establishing a correlation between the microscopic and the sedimentation sizes of anisotropic particles;

– calculation of the geometric and dynamic coefficients of the particle shape and identifying a correlation between them;

– estimating the possibility of using the variable resistance coefficient to determine the size of anisotropic particles in sedimentation.

In order to resolve the specified problems, a series of experiments was performed, in which the size of mica particles was determined by the gravitation sedimentation method in sedimentation from the starting layer, and then the same powders were analyzed with a microscope. In both cases, the mean statistical values of the weight content of mica particles for the standard fraction ranges were obtained, using a program implementing the earlier proposed method for approximating experimental data with a series of normal logarithmic laws [3].

The need to determine the geometric and dynamic coefficients of the shape of irregular particles and the relationship between them is determined by the fact that the drop in velocity of irregular-shaped particles in liquid significantly differs from the velocity of spheres of an equal weight. This difference most probably depends on the orientation of such anisotropic particles with respect to their migration direction, which is determined by the position of the center of gravity.

Speaking of the shape of particles, one usually means the conventional parameters. If particles are anisometric, certain numerical expressions can be applied to them, which determine the relationship between the typical sizes. For instance, elongated needle-shaped particles are usually estimated in terms of the ratio of their length to thickness, and flat particles are estimated in terms of the ratio of their least thickness to width. The ratio of any linear size to the least size is usually called the shape factor. However, it is believed [4] that the shape factor can be used only for qualitative estimates.

It should also be added that as the particle size decreases, the numerical expression of the shape factor becomes difficult. Thus, contrary to the widespread opinion that the anisometry of a particle decreases in crushing, it is reported in [5] that graphite with a particle size below 0.5 mm is characterized by a more laminar, irregular shape, which is due to the laminar structure of the hexagonal crystalline lattice of graphite.

Figure 1 shows the mean integral and differential distribution curves for the same mica composition, which were obtained by the layer sedimentation method and in the microscopic analysis.

The obtained cumulative curves were used to select the same pass values for which the respective sedimentation and microscopic diameters were determined. Next, a relationship between the sedimentation and the microscopic diameters was constructed, which was described by a straight-line equation (Fig. 2). The samples in the experiments were two different mica compositions of coarse and fine milling. Thus, for the finely pulverized mica (curve *1*) used in the cosmetics industry as a decorative cosmetic component this relationship was described by Eq. (1), and for the coarsely milled mica (curve *2*) it was described by Eq. (2):

$$d_m = 3.1758 d_s - 10.217; \quad (1)$$

$$d_m = 2.4032 d_s + 32.966, \quad (2)$$

where d_m is the microscopic diameter of a particle and d_s is the sedimentation or Stokes diameter.

Evidently, both distributions can be regarded as a boundary, i.e., Eq. (1) limits the distribution from below, and Eq. (2) limits it from above. The first distribution is characterized by the absence of coarse fractions, and the second one by the absence of fine fractions. It is logical to assume that the generalized formula for any distribution should pass through the coordinate origin, i.e., the “zero” particle visible with a microscope should coincide with the “zero” particle in

sedimentation. This could be experimentally confirmed by integrating the two specified distributions (Fig. 2, curve *3*).

The generalized dependence characterizing the correlation between the microscopic and the sedimentation diameters for mica of a mixed composition indeed passes through the coordinate origin and is described by the equation

$$d_m = 3.2569 d_s .$$

Using this relationship, it is possible to calculate from the known formulas [5] the values of the geometric and dynamic shape coefficients, and although knowing them does not provide for any quantitative estimate, they can give a general qualitative pattern.

The geometrical shape coefficient can be estimated using the ratio between the surface area of the particle and the surface area of an equivalent sphere, their volumes being equal. By equalizing the volume of a spherical particle to the volume of a disk of infinitely small thickness and knowing the relationship between the sedimentation and the microscopic diameters, one can determine the thickness of mica particles depending on their characteristic sizes, which is important for practical specialists:

$$l_m = \frac{2}{3}\frac{d_s^3}{d_m^2} .$$

The estimated values of the geometric and dynamic shape coefficient for mica are, respectively, equal to 1.56 and 5.29.

The same method was used to determine the same parameters and coefficients for wollastonite, whose particles are needle-shaped and can be represented as cylinders with a diameter significantly smaller than their length.

The sedimentation analysis of wollastonite is not difficult, since particles are easily wettable with water and have a density of 2.8 g/cm^3, which is sufficient for sedimentation in a gravitation field, only the equivalent sphere diameter needs to be converted into a parameter traditionally used in practice, i.e., the needle length or the ratio of the needle length to its thickness.

The analysis of wollastonite powders of different compositions using a VC-3 gravitation sedimentometer yielded stable reproducible results with an absolute mean-size error of 1 – 2 μm (δ_{50}).

The microscopic analysis data were used in parallel with the sedimentation data to convert the sedimentation diameter into the needle length or into the ratio l/d. The microscope studies revealed that, owing to the natural specifics of the mineral, the thickness of needles formed in destruction of wollastonite is a multiple of the minimum needle thickness, equal to 5 μm. Therefore, in addition to the standard microscope analysis, the analysis of the frequency of encountering needles with different l/d ratios depending on their thickness was performed as well (Fig. 3). This frequency distribution is well described by power equations:

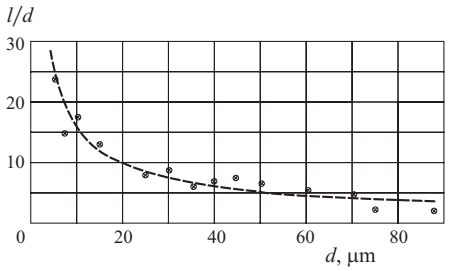

Fig. 3. Dependence of the shape coefficient of wollastonite particles l/d on their diameter d (the trend line is based on experimental data of microscopic analysis).

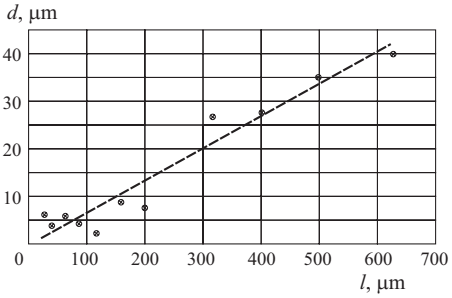

Fig. 4. Dependence of the mean diameter d of wollastonite particles on their length l (the trend line based on experimental data of microscopic analysis).

– for coarsely dispersed compositions

$$\frac{l_m}{d_m} = 22.3 d_m^{-0.4};$$

– for finely dispersed compositions

$$\frac{l_m}{d_m} = 80.5 d_m^{-0.7}. \qquad (3)$$

All further calculations were made for coarsely dispersed wollastonite, since needle-shaped particles are not typical of finely dispersed wollastonite, and their share is insignificant. The share of fine fractions significantly depends on the milling method and is especially great in mechanical milling. Therefore, it is not necessary to measure the particle length, since in this case their shape approaches the isometric shape.

The ratio between the sedimentation and the microscopic diameters, similarly to mica, is linear and can be described by the expression

$$d_m = 1.4 d_s . \qquad (4)$$

However, it is more essential for practical operators to know the particle length and the ratio of the length to the diameter.

An example of processing experimental data to determine the relationship between the mean-weighted particle diameter and the particle length is indicated in Fig. 4. As can

be seen, despite fluctuations, this relationship is virtually linear. The presence of fluctuation can be explained by insufficient representation of particles in certain size ranges based on the data of the microscopic analysis. In the trend processing (processing the curve by the least-squares method), a linear dependence was established:

$$d_s = 0.067l$$

with an approximation confidence value of 0.9279. This simple relationship between the particle length and its sedimentation diameter makes it possible to establish the relationship between the particle length and its microscopic diameter using Eqs. (3) and (4). It should be noted that in this case we determine not the real, but the most probable length with respect to the mean weighted value:

$$l_m = 80.5 d_m^{-0.7} d_m = 80.5 d_m^{0.3},$$

or in logarithmic coordinates

$$\log l_m = \log A + 0.3 \log d_s,$$

where $\log A = \log 80.5 + 0.3 \log 1.369 = 2.0422$; then

$$\log l_m = 2.04 + 0.3 \log d_s.$$

Thus, by combining two methods for determining the granulometric composition (microscopic and sedimentation), it was possible to establish a simple linear relationship between the sedimentation and the characteristic diameters in materials with anisometric shapes of particles, such as muscovite mica and wollastonite.

The existence of a simple correlation between the Stokes and the characteristic diameters for thin plates and filaments made it possible to theoretically estimate and use in practice such important characteristics of disperse anisometric materials as the ratio of the needle length to its diameter and the ratio between a thin plate thickness and its characteristic sizes.

In order to determine the granulometric composition of anisometric particles of mica and wollastonite, whose analysis by direct and indirect methods is difficult to implement due to their particle shape, one can use the layer sedimentation method, which ensures reproducible results, where the the maximum absolute error does not exceed 1 – 2 μm of the mean value and the relative error is 3%.

REFERENCES

1. P. A. Kouzov, *Principles of Analysis of Dispersion of Industrial Dust and Milled Materials* [in Russian], Khimiya, Leningrad (1987).
2. N. G. Kvesko and A. T. Roslyak, “A weighing sedimentometer for automated measurements of the granulometric composition of powders,” *Zavod. Lab.*, No. 7, 37 – 40 (2000).
3. N. G. Kvesko, “Analytical representation of multimodal functions of polydisperse material distribution,” *Sib. Fiz.-Tekh. Zh.*, No. 3, 78 – 80 (1992).
4. G. S. Khodakov, *Main Principles of Dispersion Analysis of Powders* [in Russian], Gosstroiizdat, Moscow (1968).
5. Z. R. Gorbis, *Heat Exchange and Hydromechanics of Disperse Through Flows* [in Russian], Énergiya, Moscow (1970).